Influence of RF-magnetron Sputtering System Parameters on the Process of Thin Films Nanostructure Formation

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***Abstract* — Effect of a radio-frequency magnetron sputtering system parameters on the process of thin films structure formation was analyzed. Dependence of energy delivered to the growing film by bombarding ions on the magnetron parameters and configuration was studied. Main parameters determining this energy are plasma potential, substrate ion current density, substrate bias and deposition rate. It was shown, that energetic conditions sufficiently influence on the structure formation of textured coatings. The influence of discharge gap distance and substrate bias potential on formation of hafnium diboride films structure was studied.**

Keywords — RF-magnetron; sputtering; energy; substrate bias; discharge gap; nanostructure; hafnium diboride.

I. Introduction

Magnetron sputtering is a well-known PVDs method used to produce high-tech nanoscale coatings [1-3]. Although this method has been known for a long time, it hasn`t lost its relevance now due to its advantages [2, 4]: high deposition rate; opportunity to sputter any compounds, including conductors and insulators; film's high purity and uniformity in thickness, independently on the substrate area; low-temperature plasma of magnetron discharge allows to deposit coatings on temperature-sensitive substrates; high adhesion of films; good controllability and stability of the deposition process.

The main parameters controlling magnetron sputtering process are: type of power supply (direct current supply (DC), RF-generator, pulse current source); power what supplied to the magnetron P or if more correctly – power density (power P per unit of the magnetron target surface area); working gas pressure (usually argon) p_{Ar}; substrate-to-target distance $d_{s\text{–}t}$; substrate temperature T_s; bias potential applied to substrate U_s. Besides sputtering process is determined by plasma discharge parameters, in particular, by the average plasma potential U_p. Plasma potential depends on power that injected into the discharge P, substrate bias U_s [5, 6], and also strongly depends on the design features of magnetron sputtering system: a ratio of the target area to the summary area of the all remaining surfaces that contact with discharge [5] and electrical properties of this surfaces (insulator or conductor) [7].

The energy of the target material sputtering process is determined by the difference between the plasma potential U_p and displacement potential applied to magnetron U_d in case of DC-magnetron sputtering or negative self-bias potential U_{sb} that appears on target in case of RF-magnetron sputtering [5, 8-10].

The energy of deposition process – is total energy delivered to the growing film – is determined by the following components: thermal component, exothermic and radiation components, and the kinetic energies of accelerated ions and neutral atoms, that bombarding the substrate [11, 12]. Depending on an amount of energy that delivered to growing film, coatings could formed in different structural states from amorphous-like to nanocrystalline, which respectively determines their physical and mechanical properties [13, 14].

At the low-temperature plasma conditions, which occur in magnetron sputtering, the main contribution to the total energy delivered to the growing film is made by the thermal component (energy supplied to the film by substrate heating) and the energy of the bombarding ions. Last is considered as the energy delivered to the film by bombarding ions per one deposited atom [15] or per unit volume of the film [16].

An amount of energy density delivered to the growing film by positive ions is defined by formula [16]:

$$\epsilon_b = j_s(U_p - U_s)/a_D \qquad (1)$$

where U_p – plasma potential, U_s – substrate potential, j_s – ion current density, $a_D = h/t$ – deposition rate, here h – thickness of obtained film, t – deposition time.

Thus, energy delivered to the film by bombarding ions, is determined by the following parameters: average plasma potential U_p and substrate surface potential U_S; ion current density j_s on the substrate; growth rate a_D. Ion current density j_s is a function of U_s: $j_s = f(U_s)$ and determined by substrate current-voltage characteristics and depends on magnetron sputtering system (MSS) parameters. Deposition rate a_D depends on power, that injected into the discharge, distance between substrate and target, working gas pressure, and also

U_s. In this way, three parameters included in equation of energy density ϵ_b: U_p, j_s and a_D depend on U_s, i.e. ϵ_b is a complex function of U_s depended on the properties of MSS.

Study the influence of the RF-magnetron sputtering system parameters on the process of the film nanostructure formation (by an example of HfB_2 coatings) is the aim of this work.

II. EXPERIMENTAL METHODS

A horizontal RF-magnetron sputtering system based on planar magnetron films, designed to use standard round targets with a diameter 120-125 mm and thickness up to 10 mm, was used in this work for the deposition of HfB_2. A schematic diagram of the sputtering unit is shown in Fig. 1. The magnetic field of the magnetron with intensity about 4×10^3 A/m on the surface of the target has been created by a set of annular permanent magnets (Co–Sm) with a steel polar tip. A 13.56 MHz generator with regulated power up to 1 kW is used as RF-power source. RF-generator is connected to the load by means of a matching network (L-circuit of tunable reactive elements) and blocking capacitor causing an appearance of negative self-bias on the target. Floating electrostatic capacitance in the space between target and grounded shield is determined by the geometric configuration and controlled by gap distance D between the RF electrode (target) and the grounded shield (Fig. 1). This capacitance affects the effective power at the electrode and the power loss in the network. This gap distance should be less than thickness of the ion shell (cathode dark space). In this case a discharge isn't caused in the gap. However, when the gap distance decreases excessively, the capacitive losses increase, and there is a risk of breakdown (especially if the target has sharp edges and contaminants). Resistances of plasma volume and dark spaces varied with the frequency and discharge conditions are mainly depend on the type of working gas and pressure. Therefore, changing the D value in permissible limits (taking into account the minimization of losses of the power injected into the discharge), we can vary a threshold value of working gas pressure to ignite the discharge. Thus, in the case of D = 8 mm, the discharge ignites at pressures $p_{Ar} \geq 0.87$ Pa, and when D = 6.5 mm discharge ignites at pressures greater than 0.32 Pa.

The substrate holder is electrically insulated from the housing of the installation by ceramic insulators and intended for fastening plate-like substrates. A DC-voltage source connected to the substrate holder through the high-pass filter, allowing a bias potential on the substrate in the range from –100 to +100 V. The values of the bias potentials and ion currents on the target and substrate are monitored by pointer instruments (measurement error ±5%). The samples heated by means of a tungsten glowing spiral with a diameter 2 mm. The chromel-alumel thermocouple provides temperature control up to 1100 K.

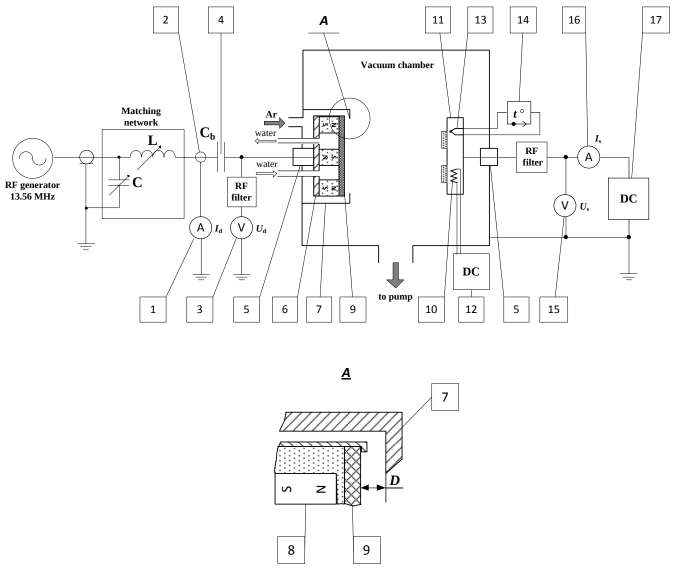

Fig. 1. Scheme of magnetron sputtering system: system: 1 – discharge current indicator; 4 2 – Rogowski coil; 3 – negative self–bias potential on target indicator; 4 – blocking capacitor; 5 – insulator; 6 – unbalanced magnetron; 7 – anode; 8 – permanent magnet; 9 – target; 10 – substrate holder heater; 11 – substrate holder; 12 – substrate holder heater`s power supply; 13 – thermocouple; 14 – temperature sensor; 15 – indicator of the substrate bias; 16 – ion current indicator through the substrate holder; 17 – DC power supply for the substrate bias application.

Sputtering of a hot-sintered target HfB_2 was carried out in a plasma of Ar^+. Hafnium diboride coatings were deposited on stainless steel substrates 12X18H10T (20×10 mm) pre-polished to roughness R_a = 0.25 μm. The residual pressure in the chamber before deposition was $2\div3\times10^{-3}$ Pa. Working gas pressure of 0.87 Pa, RF–generator power of 500 W and the substrate temperature of ~400°C were maintained during deposition. The bias potential applied to the substrate was varied from +50 to –50 V relatively to the ground in 25 V increments. Substrate-to-target distance was d_{s-t} = 60 mm.. The deposition time was 60 min for all modes. An average thickness of HfB_2 coatings under these conditions was ~1 μm.

X-ray diffraction researches of the material structure were carried out using automated diffractometer DRON-3. The CuKα radiation (wavelength of 0.154 nm) and the Bragg-Brentano focusing method θ – 2θ (2θ – Bragg angle) were used in the shooting. The values of current and voltage on the X-ray tube were 20 mA and 40 kV. Shooting of specimens was carried out with horizontal slits of 4 mm on the tube and of 1 mm on the detector in continuous registration mode with a rate of 34 1°/min in a 2θ angle range from 25° to 60°

III. RESULTS AND DISCUSSION

The relationship between substrate ion current density j_s and bias potential U_s is determined by current-voltage characteristic measured on the substrate. Fig. 2 exhibits *I-V* characteristic measured on substrate for two gap values D between target and grounded shield D = 6.5 mm and D = 8 mm.

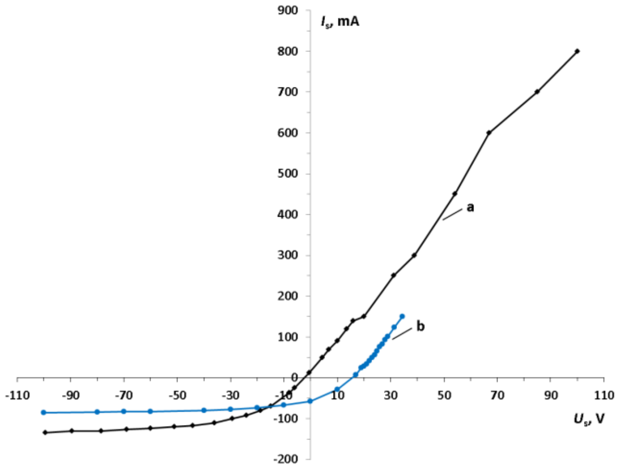

Fig. 2. Current-voltage characteristics measured on the substrate at different gap distances: a) D = 6.5 mm , b) D = 8 mm.

Comparing the current-voltage characteristics, we can see that dependence doesn't change qualitatively, but the characteristic is shifted to the right. Accordingly, the floating U_f and plasma U_p potentials increase, the saturation ion current also changes. Finally it leads to new energy distribution. Figures 3, 4, 5 exhibit diffraction patterns of films obtained at different discharge gap distances D and substrate bias potential varied from –25 V to +25 V.

Analyzing obtained diffraction patterns we can see certain regularities of the formation of hafnium diboride films. All the main peaks (00.1), (10.0), (10.1), (00.2), (10.2) has appeared, that respond to the structural type AlB_2(space symmetry group P6/mmm).

At the negative bias potential –25 V (Fig. 3 a, b) and discharge gap values of 8 mm and 6.5 mm hafnium diboride nanocrystalline films with weak growth texture normally to the plane (00.1) were formed.

In both cases at grounded substrate holder (Fig. 4), the formation of textured nanocrystalline films with growth texture normally to the plane (00.1) also occurred. However, at discharge gap of 8 mm texture degree (00.1) decreases and the intensity of the peaks (10.0) and (10.1) increases. At discharge gap 6.5 mm the formation of strongly textured films takes place, which is confirmed by the appearance of diffraction peaks (00.1) and (00.2), the remaining peaks were absent.

At the positive substrate bias of +25 B and discharge gap of 8 mm (Fig. 5 a) nanocluster structure with preferential orientation in the direction of (10.0) was formed. In case of discharge gap of 8 mm (Fig. 5 b) the formation of a nanostructure occurred, as evidenced by diffuse peaks (00.1), (10.0), (10.1) and (00.2).

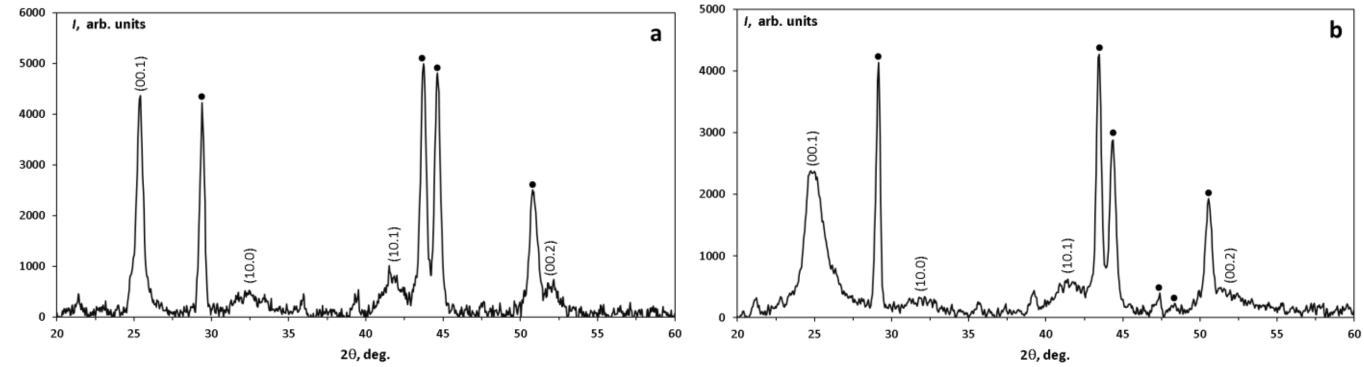

Fig. 3. Diffraction patterns of HfB_2 films obtained at substrate bias –25 V, but for different discharge gaps: a) 8 mm; b) 6.5 mm. The symbol "•" indicate the reflexes of the substrate.

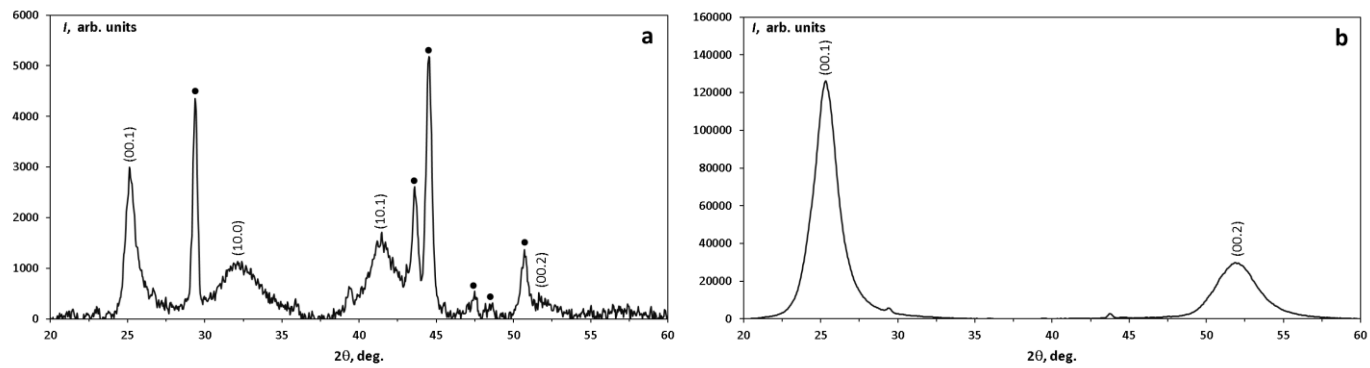

Fig. 4. Diffraction patterns of HfB_2 films obtained at substrate bias 0 V, but for different discharge gaps: a) 8 mm; b) 6.5 mm. The symbol "•" indicate the reflexes of the substrate.

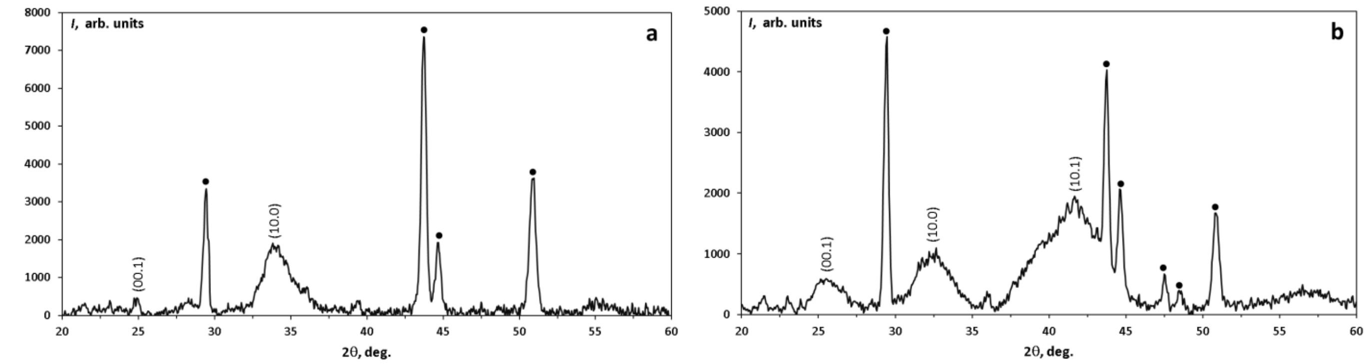

Fig. 5. Diffraction patterns of HfB_2 films obtained at substrate bias +25 V, but for different discharge gaps: a) 8 mm; b) 6.5 mm. The symbol "•" indicate the reflexes of the substrate.

Thus the structural state of the film coating is determined by the process energy and substantially changes depending on the applied bias potential and on various discharge gaps. Main factors related to amount of energy caused by particle bombardment are bias potential, ion current density and deposition rate, determined by magnetron sputtering system.

IV. CONCLUTIONS

Plasma potentials determined by the deposition parameters and the geometry of the MSS, as well as the substrate bias potential, make possible to vary the supplied surface energy, which mainly depends on the current-voltage characteristic of the substrate.

Formation of nanostructured films is deeply connected to growing energy, which is determined by the bunch of factors like plasma potentials and particle bombardment. So self-reproducibility of films with specified properties requires a precise control of each MSS parameter.

The magnetron sputtering system is a complicated combination of various parameters and configurations making it possible to influence on the energy of the obtained coatings and formation of their nanostructure (as in the case of hafnium diboride films).

This work was performed within the framework of a budget themes No. 0116U002621 "Physical bases of the formation of the structure and properties of nanostructured transition metal borides, nitrides and boridonitrides films for application in mechanical engineering" and No.0117U002247 "Technological bases of the synthesis of nanostructured, nanocomposite and multilayer coatings of refractory compounds for application in mechanical engineering".

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